

MCHM and PPH Holding Time Test – Summary

This test was performed to determine whether MCHM and PPH are stable in hexane extracts over a period of 28 days.

The procedure was performed as follows. Thirteen simulated samples, consisting of 100 mL of laboratory water, were injected with MCHM and PPH. Of the thirteen samples, four contained high concentrations of MCHM and PPH, four contained mid-levels, and four contained low levels. The thirteenth sample contained no MCHM or PPH. Fifteen grams of sodium chloride was then dissolved in each sample, followed by the addition of 2 mL of hexane. The samples were then shaken on a mechanical shaker for 30 minutes and the hexane layer was siphoned off. The hexane extract was then placed in a sealed vial. Aliquots of the hexane extract were then injected into a gas chromatograph utilizing a flame ionization detector (GC/FID) for determination of MCHM and PPH.

Aliquots were analyzed every 7 days for a total of 1 initial determination and 4 subsequent determinations over 28 days. Upon initial analysis, it was found that the four low level sample extracts responded too poorly to produce meaningful results and these four extracts were discarded from testing. A calibration curve was analyzed only with the initial determination to avoid a situation where both extracts and calibration standards would degrade at a constant rate and would thereby mask any actual degradation in the sample extracts. The calibration curve was verified by the analysis of a mid-point standard at the beginning and end of each of the four subsequent determinations after initial analysis.

Results from the holding time test displayed no significant trend with regards to either decreasing or increasing concentrations of MCHM or PPH in sample extracts over 28 days. Some minor inconsistencies in results were observed which can be attributed to normal analytical variability within the GC/FID itself.